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MEASUREMENT OF SOFT X-RAY ABSORPTION SPECTRA WITH A
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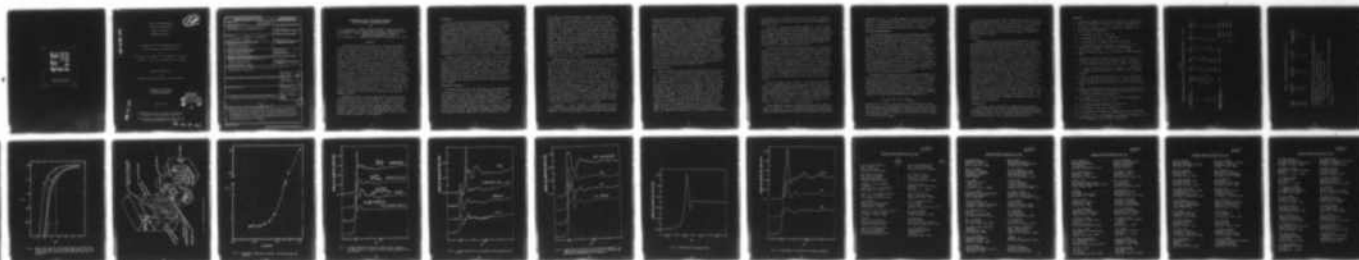
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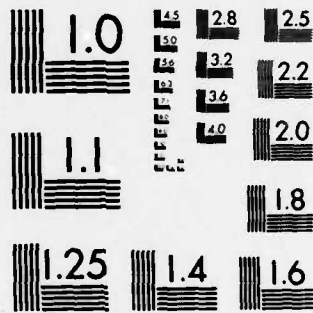
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Measurement of Soft X-ray Absorption Spectra
with a Fluorescent Ion Chamber Detector

by

F. W. Lytle, R. B. Gregor, D. R. Sandstrom, E. C. Marques,
J. Wong, C. L. Spiro, G. P. Huffman, and F. E. Huggins

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Absorption spectra at the K-edges of S, Cl, Ar, and K have been measured in fluorescence mode on the hard x-ray beam lines at SSRL using an ion chamber detector collecting 20% of 2 π . In spite of diminished x-ray flux and fluorescent yield, sufficient sensitivity was obtained to characterize one percent S in coal. On an unfocused wiggler beam line a resolution of 0.5 eV was obtained at the S K-edge.		

MEASUREMENT OF SOFT X-RAY ABSORPTION SPECTRA
WITH A FLUORESCENT ION CHAMBER DETECTOR

by

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ABSTRACT

The problem of absorption of soft x-rays by the thick Be windows in the hard x-ray beam lines at SSRL is well known. Although the signal at 2.4 keV is reduced by $\sim 10^3$ we have routinely measured the absorption spectra of S (2472 eV) and elements at higher energies including Cl, Ar and K. These spectra were obtained on beam lines II-3 (focused) and VII-3 (wiggler side station, unfocused) with Si(111) monochromator crystals. A He bag was used to enclose the end of the beam pipe, the I_0 ion chamber and the sample box. The monochromator was detuned 50 to 90% to minimize harmonics. The detector was a drum-shaped ion chamber similar to that described by Stern and Heald (1) located at right angles to the beam path with a solid angle of acceptance of 20% of 2π (front side collection). Although the diminished x-ray flux and decreasing fluorescent yield are serious negative factors at these low x-ray energies the spectra from thick samples contained in 6 μ m polypropylene bags are of excellent quality with sufficient sensitivity to characterize one percent S in coal. The side station spectra (no focusing mirror) have excellent resolution, $\Delta E = 0.5$ eV at the S edge. The practical advantages of sample handling on these beam lines compared to JUMBO are obvious.

Representative spectra are shown comparing data from focused and unfocused beam lines and with S data from JUMBO (2). Comparison of Ar and KCl data to excellent data found in the older literature (3,4,5) allow a confirmation of the resolution function (energy bandpass) of the monochromator. A simple new beam line is suggested which would allow a substantial increase in low energy x-ray flux (measurements down to Al and Si) with the sample and detector in a He atmosphere.

INTRODUCTION

On hard x-ray beam lines at synchrotron radiation sources thick Be windows are used in order to secure the vacuum requirements of the storage ring. Additional absorbers in the beam may be 25 μm of pyrolytic carbon (to absorb heat) and a Kapton window after the x-ray monochromator. All of these absorb soft x-rays with a sharp cut-off between 2 and 4 keV. Figure 1 illustrates the case of typical beam lines at Stanford Synchrotron Radiation Laboratory (SSRL). Curve B illustrates the case for a wiggler side-station (beam lines IV-1, IV-3 and VII-3) with 533 μm Be, 35 μm C and 1 m He along the beam path. Curve C illustrates the effect of an additional 9 m of He which is typical of beam lines I-5, II-2 and II-3. This 9 m of He decreases flux by factors of 2 - 3 at the lower energies. Curve A is a hypothetical case with 125 μm Be and 25 μm C which will be discussed later.

Although the transmitted intensity is falling very rapidly and is down by $\sim 10^3$ at the energy of the S K-edge [The energies of the S (2472 eV) and K (3607 eV) edges are indicated on the graph.] we were able to obtain good data for S, Cl (6) and the higher Z elements on these beam lines. These elements also may be examined on JUMBO (2); however, it is a particular advantage to be able to use more available beam lines and not be bothered by the high vacuum requirements of JUMBO. Herein we discuss our experimental technique, the x-ray detector, a variety of data for S, Cl, Ar and K and make comparisons between data from beam lines at SSRL and with data in the literature.

EXPERIMENTAL TECHNIQUE

We used the standard two-crystal monochromator (Super Monochromator) at SSRL with Si(111) crystals, a 1 mm (vertical dimension) entrance slit and a He path enclosing the end of the beam pipe, scatter slits, I_0 detector (standard 15 cm parallel plate ion chamber) and the sample area. Windows on the I_0 detector were replaced with 6 μm polypropylene (7). The fluorescent x-ray ion chamber detector is shown in figure 2. This unit is derived from that described by Stern and Heald (1). The sample is located at 45° to the x-ray beam. Fluorescent and scattered x-rays emerging from the surface of the sample impinge upon a filter (typically a Z-1 filter)

which removes the scattered radiation and passes the fluorescent x-rays from the sample. The slit assembly is oriented as shown so that radiation diverging from the sample position passes through the slits unimpeded. The purpose of the slits is to (partially) reject the fluorescent radiation arising in the filter material by restricting the angular aperture for this radiation. It was reported (1) that the slits reduced the fluorescence from the filter by a factor of 8. The outer window of the ion chamber is 6 μ m aluminized Mylar with a center electrode and back plane of 90% transparent Ni mesh. The outer electrodes are operated at -45 V and the photoelectron current collected on the center electrode and amplified to a positive d.c. voltage typically between 0.1 - 1.0 V. This is connected to an analogue-to-digital converter and scaled in the same way as the I_0 detector. The active diameter of the ion chamber is 8.25 cm with a mid-sample to ion chamber distance of \sim 5.5 cm. This results in a collection efficiency of \sim 20% of 2π from the front face of the sample.

Depending upon the element of interest various gases (or mixtures) may be used in the ion chamber. The collecting electrode is centered 1.5 cm from the aluminized Mylar front window and Ni mesh back plane. The maximum signal will be detected if the x-rays can be mostly absorbed within the body of the ion chamber, i.e., with a mean free path for absorption of 1-2 cm. Table I lists the calculated properties of various ion chamber gases. For S, Cl and Ar spectra nitrogen was used in the fluorescent detector; for K spectra an available mixture of 10% He, 90% Ne was used. In all cases He was used in the I_0 detector. The powdered samples and NaCl solution were contained in 6 μ m polypropylene bags (7). For the Ar experiment a small amount of Ar was mixed with the He purging the sample box. No scattered radiation filter was used for any of the measurements. For elements with $Z < 23$ (V) the $Z-1$ rule no longer holds, i.e., the $Z-1$ absorption edge does not fall in the energy range between the K_{α} lines and K-absorption edge of element Z . In some cases it may be possible to use elements with suitable L-edges as filters for low Z elements (e.g., L_{111} for Ti K-edge).

Although the (222) reflection is forbidden from Si(111) the fully tuned beam from the monochromator was primarily composed of higher harmonics. In order to even observe an absorption edge it was absolutely

essential to remove the harmonics from the beam. This was conveniently done by detuning (8) the monochromator so that the diffracting planes of the two crystals were not precisely parallel. This procedure selectively removes higher order reflections (8). Figure 3 illustrates the detuning procedure for S. The monochromator was adjusted to an (indicated) energy position below the S edge. At this position only the harmonic contaminants in the beam have sufficient energy to excite K-edge fluorescent x-rays from S. When the normalized signal from the fluorescent ion chamber, I_F/I_0 is plotted versus degree of detuning, $I_0/I_0(\text{max})$, where $I_0(\text{max})$ is the highest observed signal from the I_0 detector, the curve decreases asymptotically to a horizontal line at $\sim 80 - 90\%$ detuning (lesser detuning for higher energies). At this point the beam has negligible harmonic content; I_F is proportional to I_0 , and is due to scattered radiation of the desired energy. The onset of S K-fluorescence excited by harmonics creates the steep rise in the curve and was found to be a sensitive indicator of beam harmonics. This technique of detuning can be done very quickly without additional energy sensitive detectors and also may be easily and frequently checked.

ABSORPTION EDGE DATA

Here we illustrate a variety of absorption edge data for S, Cl, Ar, and K in order to illustrate the performance of the spectrometer and to show that the sensitivity of detection and the resolution is sufficient for quality work. With the exception of the KCl (focused data) in figure 6, all data were obtained on beam line VII-3. The S K-edge data in figure 4 illustrates the sensitivity of the x-ray absorption near edge structure (XANES) to the bonding environment of S in various organic compounds. The zero of energy was chosen as the 1st inflection point for elemental S. Each compound is identified on the graph. The data show a remarkable sensitivity to the chemical environment of S. In figure 5 data from S in Illinois No. 6 Coal (1.2 wt.% S) and possibly related reference compounds are shown. Since our data base of S reference compounds was small our identification must be tentative; however, a significant fraction of the S in Illinois No. 6 appears to be in a mixture of sulfate and pyrite (or marcasite) environments. For comparison, in Hussain, et al. (2) the spectrum for subbituminous

coal (figure 18) is very similar to our Illinois No. 6 coal. We have also recently identified a Winifrede seam coal as containing thianaphthene and pyrite-like S (9).

On the basis of comparing the sulfate-like peak at ~ 10 eV our resolution appears to be significantly better than JUMBO (2 eV versus 3 eV FWHM) as it should be considering that a focusing mirror and Ge(111) crystals were used on JUMBO. The sharp little spikes appearing on the curves in figures 4 and 5 are diffraction peaks from the polycrystalline powder samples which enter the detector. In general, they could be minimized by grinding to a finer texture or eliminated with a suitable filter.

In figure 6 a variety of Cl K-edge data are compared. In the top two curves a ~ 1 M NaCl-in- H_2O solution is compared with polycrystalline NaCl. In the bottom two curves data for polycrystalline KCl is compared from the wiggler side station, line VII-3, and from the focused line, II-3. Note the serious degradation in resolution on the focused line although Si(111) crystals were used with each monochromator. This is due to the mixing of horizontal and vertical divergence by the focusing mirror. The high resolution KCl data is nearly an exact copy of that of Parratt and Jossem (5). Also note that the same observation applies to the K K-edge data for KCl in figure 8. It is remarkable that the Cl XANES out to 20 eV is so different in such similar compounds electronically and structurally as NaCl and KCl.

The Ar gas K-edge is shown in figure 7. This spectrum is a classic test of x-ray spectrometry and was first measured at high resolution by Parratt (3) and later remeasured by Schnopper (4). Both used high resolution monochromators operated in the (1, +1) dispersive mode and equipped with cleaved, polished, and etched calcite crystals. A numerical resolving power, $\lambda/\Delta\lambda \approx 10,000$, was claimed (5). The uncorrected FWHM of the first peak was ~ 0.8 eV as compared to ~ 1 eV for the data in figure 7. The resolution of the spectrometer will be discussed later.

The K K-edge data in figure 8 illustrate the sensitivity of XANES to three K environments. Illite is a hydrated aluminosilicate mineral having a sheet structure composed of two layers of SiO_4 tetrahedra and one central

octahedral AlO_6 layer. K is located between the SiO_4 layers of two adjacent sheets and is 12-coordinated by oxygen. KCl has a cubic structure in which each atom is surrounded by six of the opposite kind. In KC_8 K is intercalated into graphite between two C_6 rings with 12 C 1st neighbors.

RESOLUTION OF MONOCHROMATOR

It has been pointed out by Brown (10) and by Kostroun and Materlik (8) that with existing synchrotron sources the energy resolution is usually determined by the divergence of the emitted x-rays, not by the diffraction width of the monochromator crystals. This was not generally true for our experiments. The 1 mm entrance slit fixed the vertical divergence at 5×10^{-5} radians which was a smaller divergence than that accepted by $\text{Si}(111)$ at these low x-ray energies. Various energy band widths are estimated in table II. δE is the width due to the diffraction profile (11), ΔE assumes a constant 1 mm slit and $\Delta E'$ is the calculated divergence for a wiggler line with electron energy of 3 GeV and wiggler field of 18 KG. The divergence from a SPEAR bending magnet is 1.2×10^{-4} radians (10). It is obvious that below 5 keV the crystal diffraction pattern dominated the energy band width. At the lowest energies the slit could have been opened to 3 mm for more intensity without degrading the energy resolution.

The resolution of two-crystal monochromators has been discussed (12,13). The total band pass of the spectrometer is a convolution of the divergence of the source and the width of each diffracting crystal. It also has been demonstrated that measured spectral data may be deconvolved provided that each component of the total resolution is accurately known (14). Since this is not generally the case a useful estimate of the width of the total instrument function may be made by assuming that every resolution component is Gaussian in shape, hence the components add in quadrature (13).

$$\Delta E_{\text{Total}}^2 = \delta E_1^2 + \delta E_2^2 + \Delta E_{\text{Source}}^2$$

where δE_1 and δE_2 refer to the two crystals, which are identical in this case, and ΔE_{Source} is the divergence of the emitted x-ray beam (here limited by a 1 mm slit). This approximation is certainly contrary to what is known about the actual shape of δE and ΔE but is used here (as it was in the past) as a rough estimate of ΔE_{Total} .

The Ar and KCl data provide a basis for comparison with very well characterized monochromators described in the literature (3,5). These instruments used calcite crystals with a numerical resolving power $\lambda/\Delta\lambda \approx 10,000$ or an energy band width of $\Delta E \approx .3$ eV at the Ar K-edge. The relative quality of our data and theirs suggests that our estimate of ΔE_{Total} is approximately correct as given in table II.

DISCUSSION

The feasibility and ease which we have demonstrated here in measuring the K-edge spectra of S under non-vacuum conditions should open up new x-ray spectroscopic investigations of the bonding and structure of S-containing metallo-proteins and other biological materials that are usually vacuum incompatible or unstable. The sensitivity of the fluorescent x-ray detector was a surprise considering that the fluorescent x-ray yield is so low, e.g., .07-to-.13 for S-to-K (15). The explanation lies in the high incident flux and the large solid angle of detection. The qualitative success and relative simplicity of these experiments relative to the severe requirements of a high vacuum environment suggest that new beam lines should be implemented. The excessive thickness of the present Be windows is required not just for a vacuum barrier but also due to the thermal load and possible erosive chemical reactions induced by the x-ray beam. Simply placing the equivalent of the existing monochromator in the vacuum with a thin Be window in the monochromatized beam would result in the spectrum shown in figure 1A. X-ray intensity equivalent to that now realized for S would extend to the Al K-edge at 1.56 keV. The flux at the S K-edge would be 10^2 higher. The end station could be a simple He-filled box with easy access for samples. Si(111) crystals would allow examination of the P K-edge. Some other crystals such as InSb(111) (2) and YB₆₆ (400) (16) would be required for Si and Al. This suggested beam line would bring a new degree of experimental ease to these frequently occurring elements.

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11. We thank Zofia Rek, SSRL, for calculations of the Si(111) Darwin widths in table II. At 8.05, 4.97, 3.20 and 2.47 keV the FWHM are 7.52, 11.6, 21.1 and 35.8 seconds of arc, respectively.
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TABLE I

PROPERTIES OF GASES FOR X-RAY DETECTOR, CALCULATED MEAN FREE PATH ($I/I_0 = 1/e$), cm

ENERGY, KeV	He	N	Ne	Ar	Kr	Xe
2	887	1.6	.9	1.1	.06	.08
3	3589	5.4	2.7	3.3	.1	.2
5	21459	26.1	12.2	1.3	.8	.3
8	114×10^3	117	50.9	4.7	2.8	.6
10	250×10^3	241	102	8.8	5.2	1.0
15	etc.	910	366	29	2.3	3.0
20		2356	916	70	4.9	6.8
ABSORPTION EDGE(S), eV	24.6	401.6	866.9	3202.9	14325.6	34561.4
					L_I 1921.0	5452.8
					L_{II} 1727.2	5103.7
					L_{III} 1674.9	4782.2

TABLE II. CALCULATED ENERGY BAND WIDTHS FOR Si(111)

E, eV	$\delta E, \text{eV}^{(a)}$	$\Delta E, \text{eV}^{(b)}$	$\Delta E', \text{eV}^{(c)}$	$\Delta E_{\text{Total}}, \text{eV}^{(d)}$
2472	.32	.09	.8	.5
3203	.41	.20	1.5	.6
4966	.65	.57	3.5	1.
8048	1.14	1.6	7.7	2.

(a) ESTIMATED FROM CALCULATED Si(111) SINGLE CRYSTAL DIFFRACTION PROFILE (9).

(b) ASSUMES CONSTANT 5×10^{-5} DIVERGENCE THROUGH 1 mm SLIT.

(c) ASSUMES FULL DIVERGENT BEAM FROM WIGGLER, FWHM = 2.2σ ,

$$\sigma = \frac{1}{\gamma} (E_c/3E_x)^{1/2} \text{ where } \gamma = .170 \text{ milliradian,}$$

E_c = critical energy ≈ 10 keV and E_x is the energy of the emitted x-rays

$$(d) \Delta E_{\text{Total}}^2 = 2 \delta E^2 + \Delta E^2$$

- Fig. 1. Spectra calculated for A) 125 μm Be and 25 μm C, B) 533 μm Be 35 μm C and 1 m He (This is typical of SSRL wiggler beam lines.), and C) 533 μm Be, 35 μm C plus 9 m of He (SSRL beam lines I-5, II-2 and II-3).
- Fig. 2. Fluorescent x-ray ion chamber.
- Fig. 3. The effect of detuning on intensity. The text describes the procedure.
- Fig. 4. S K-edge spectra for a variety of organic sulphur compounds. The zero of energy is taken at the first inflection point of pure S at 2472 eV.
- Fig. 5. S K-edge spectra for some inorganic sulphur compounds and a coal.
- Fig. 6. K-edge spectra for chlorine in the indicated compounds. The lower curve was obtained on beam line II-3 in focusing mode and shows a large degradation of resolution.
- Fig. 7. K-edge spectra for gaseous argon.
- Fig. 8. K-edge spectra for potassium in selected compounds.

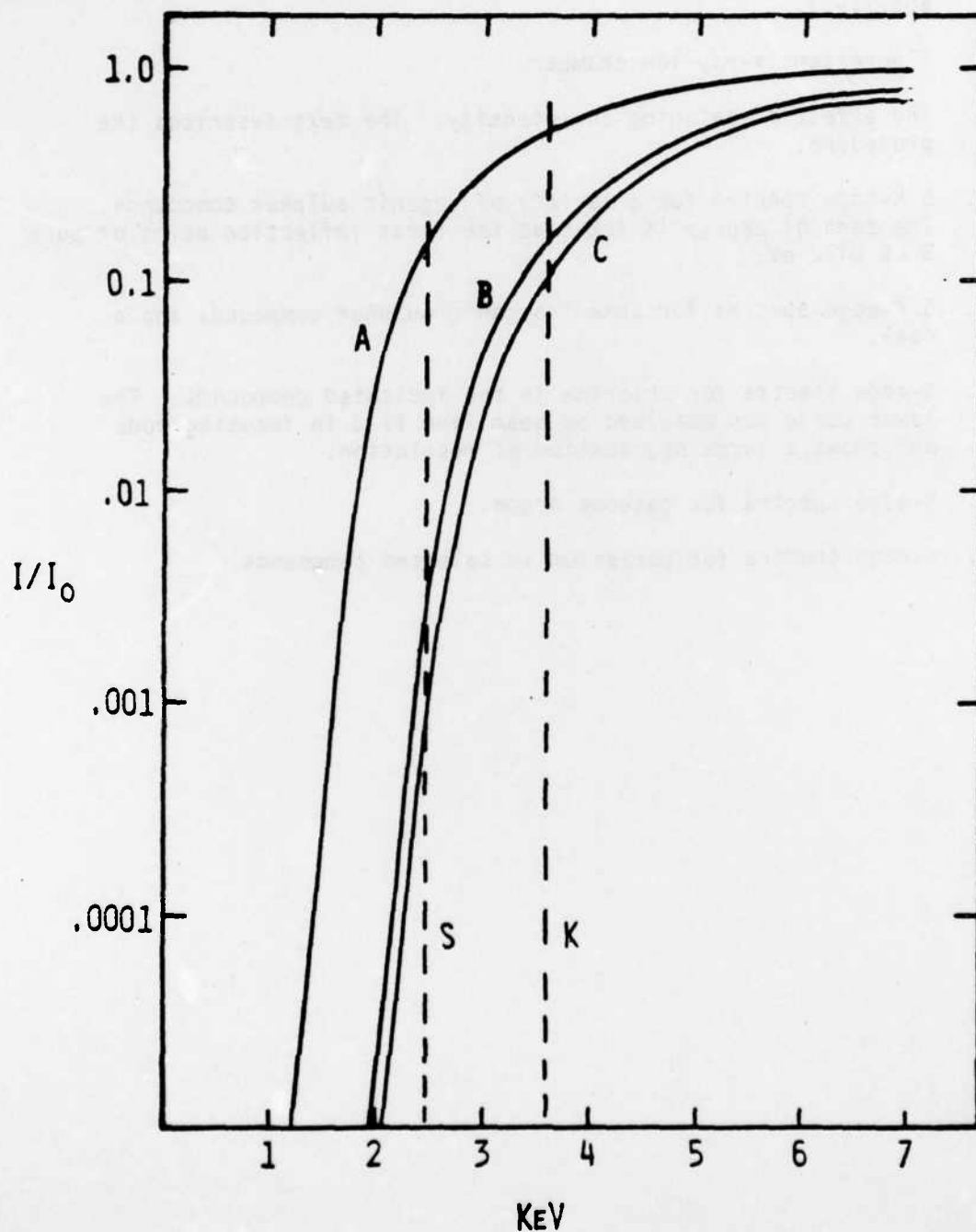


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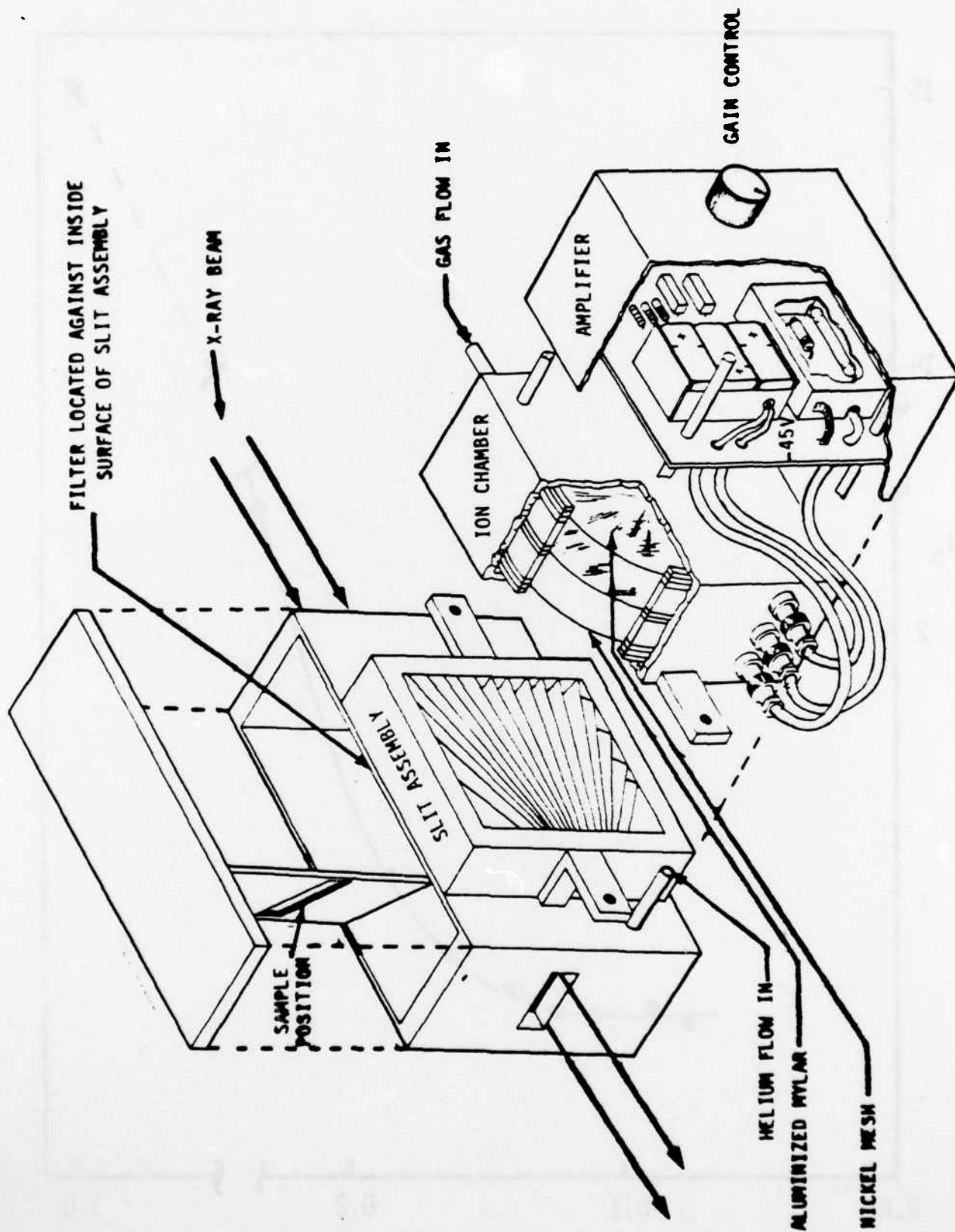


Fig. 2. Fluorescent x-ray ion chamber.

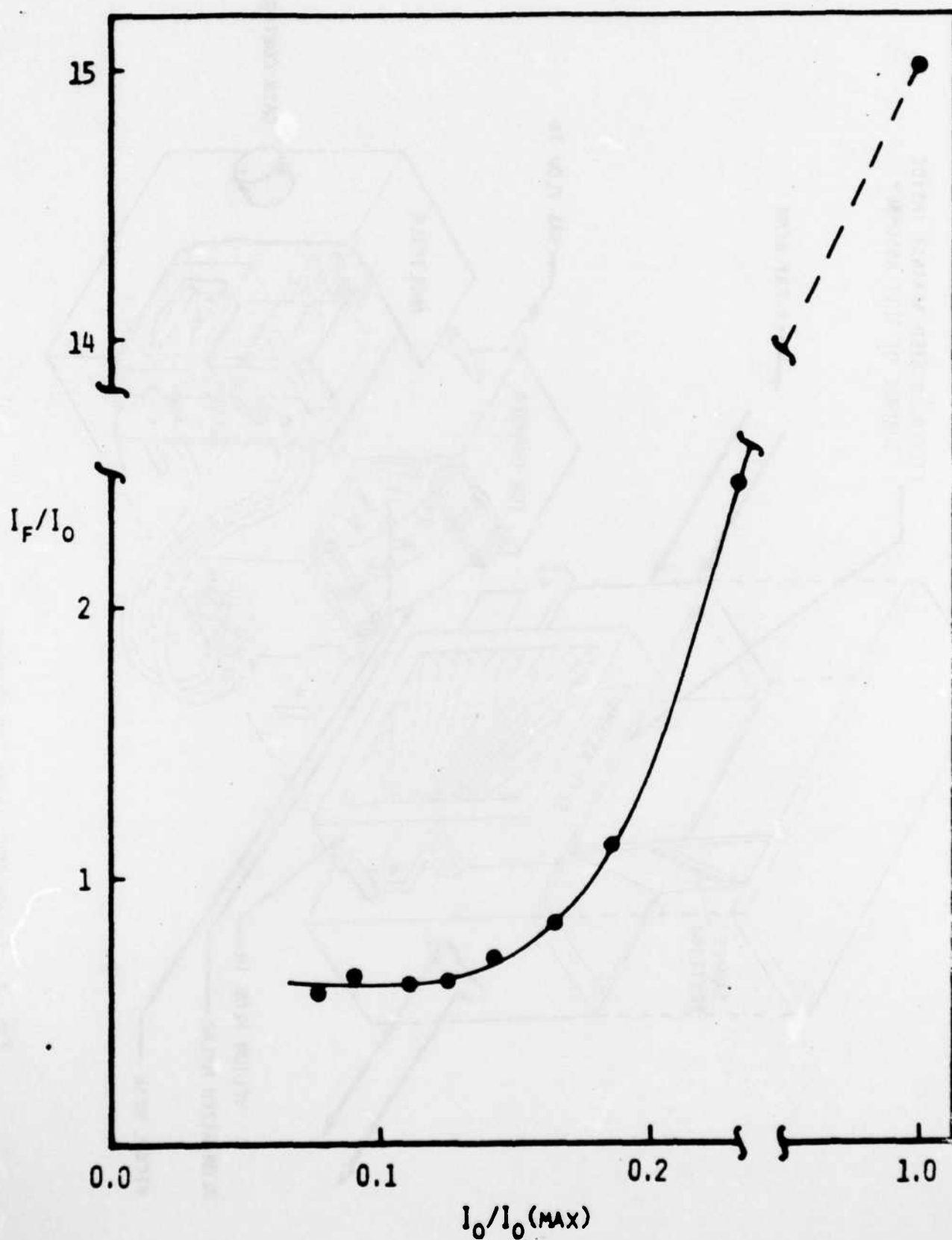


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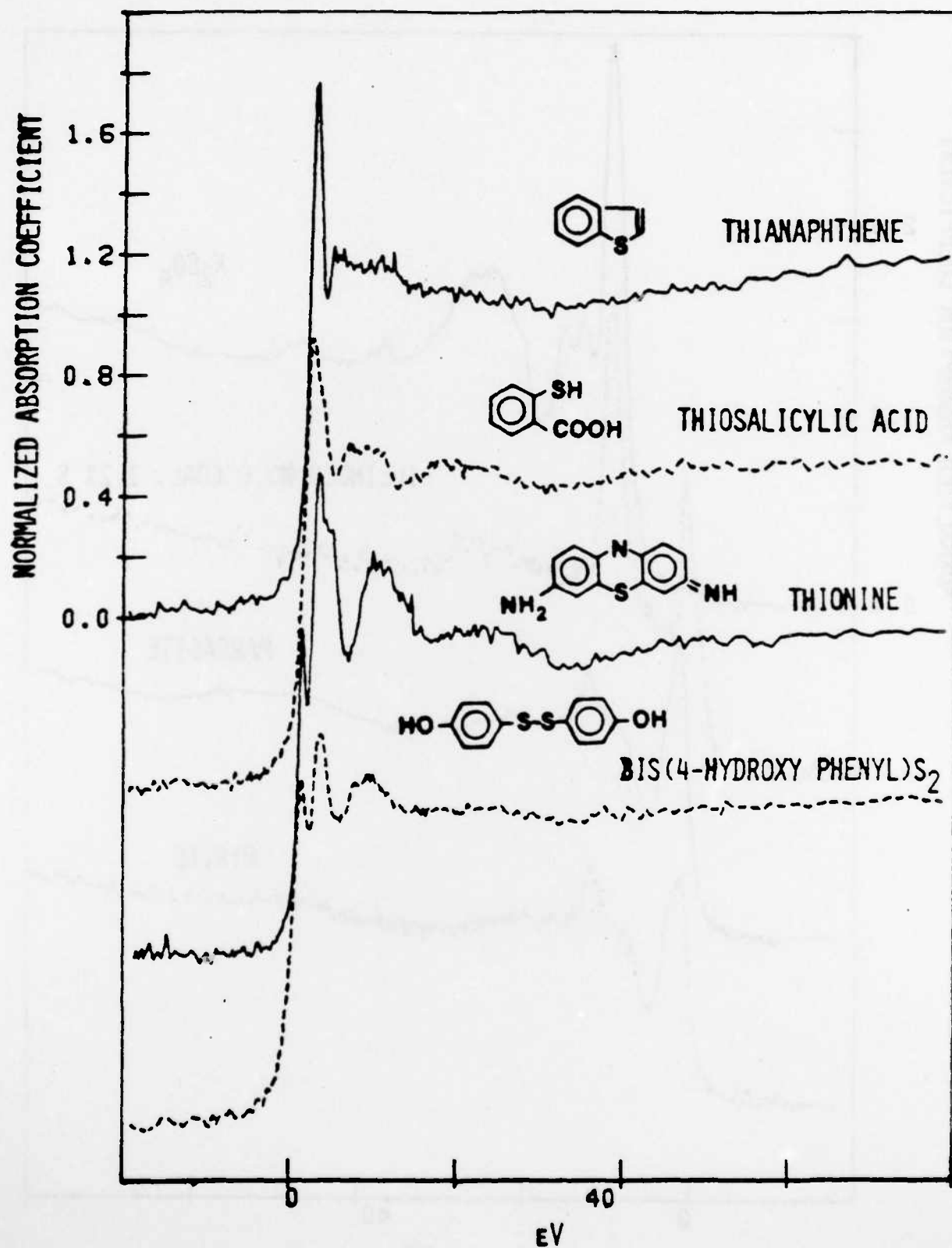


Fig. 4. S K-edge spectra for a variety of organic sulphur compounds. The zero of energy is taken at the first inflection point of pure S at 2472 eV.

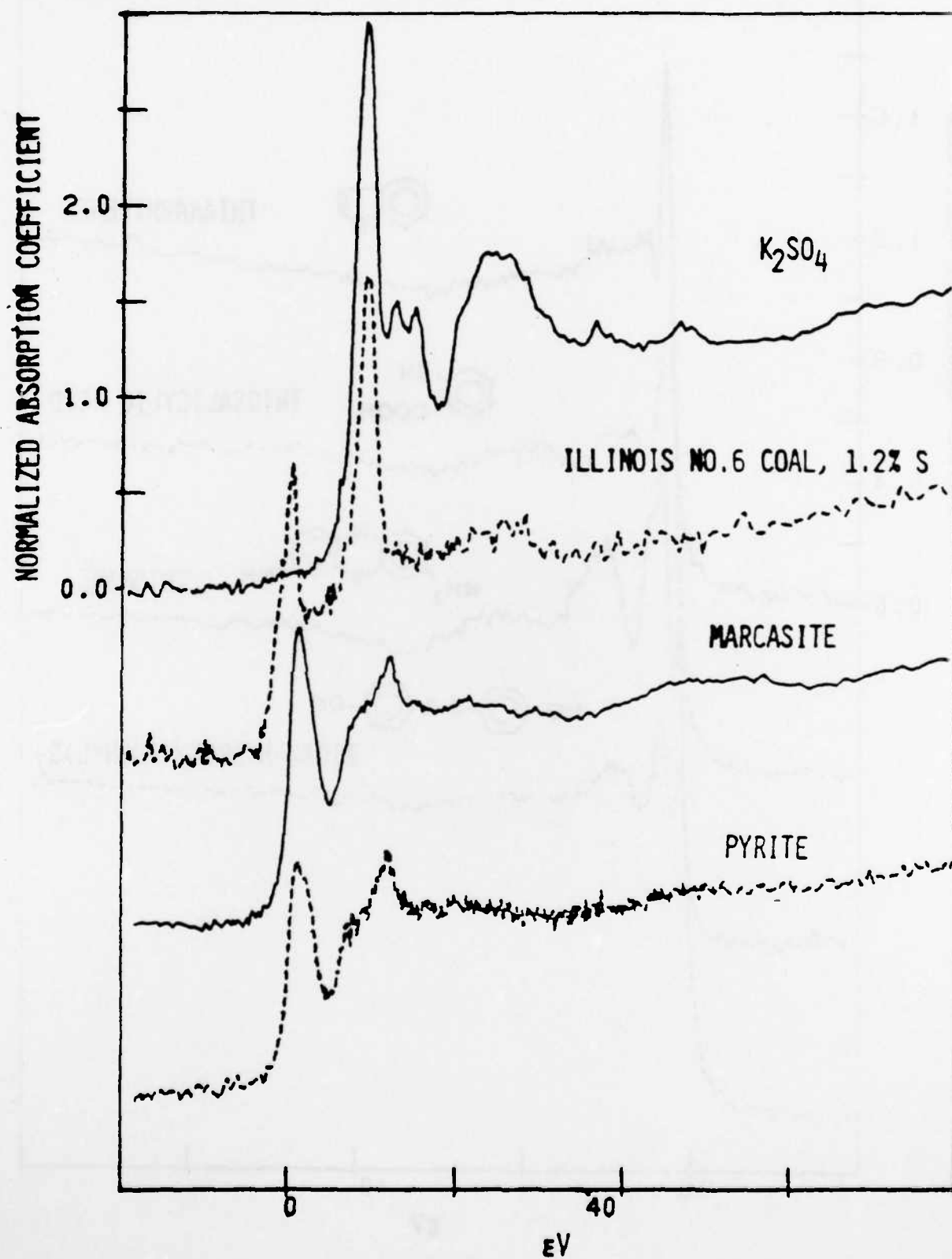


Fig. 5. S K-edge spectra for some inorganic sulphur compounds and a coal.

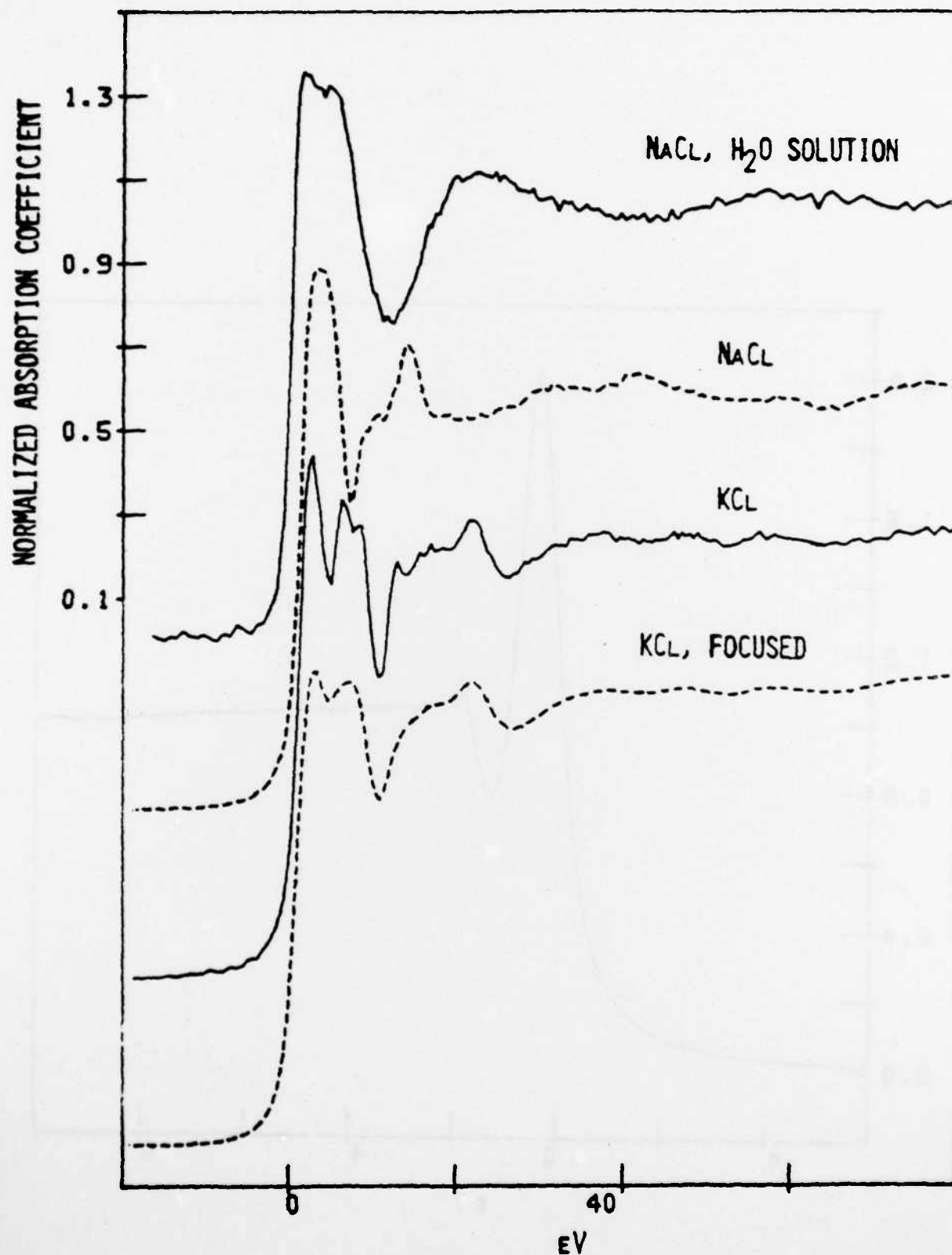


Fig. 6. K-edge spectra for chlorine in the indicated compounds. The lower curve was obtained on beam line II-3 in focusing mode and shows a large degradation of resolution.

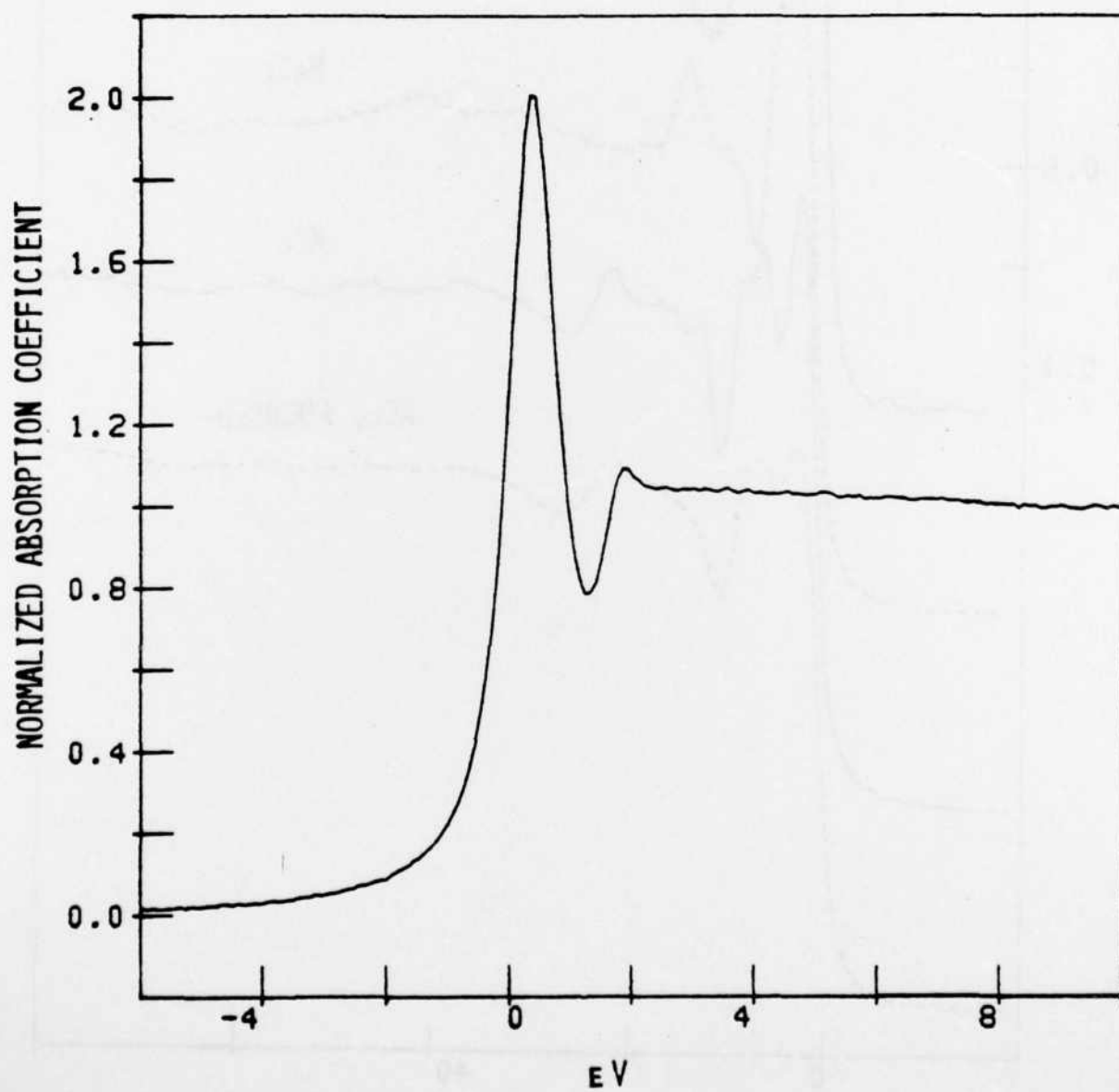


Fig. 7. K-edge spectra for gaseous argon.

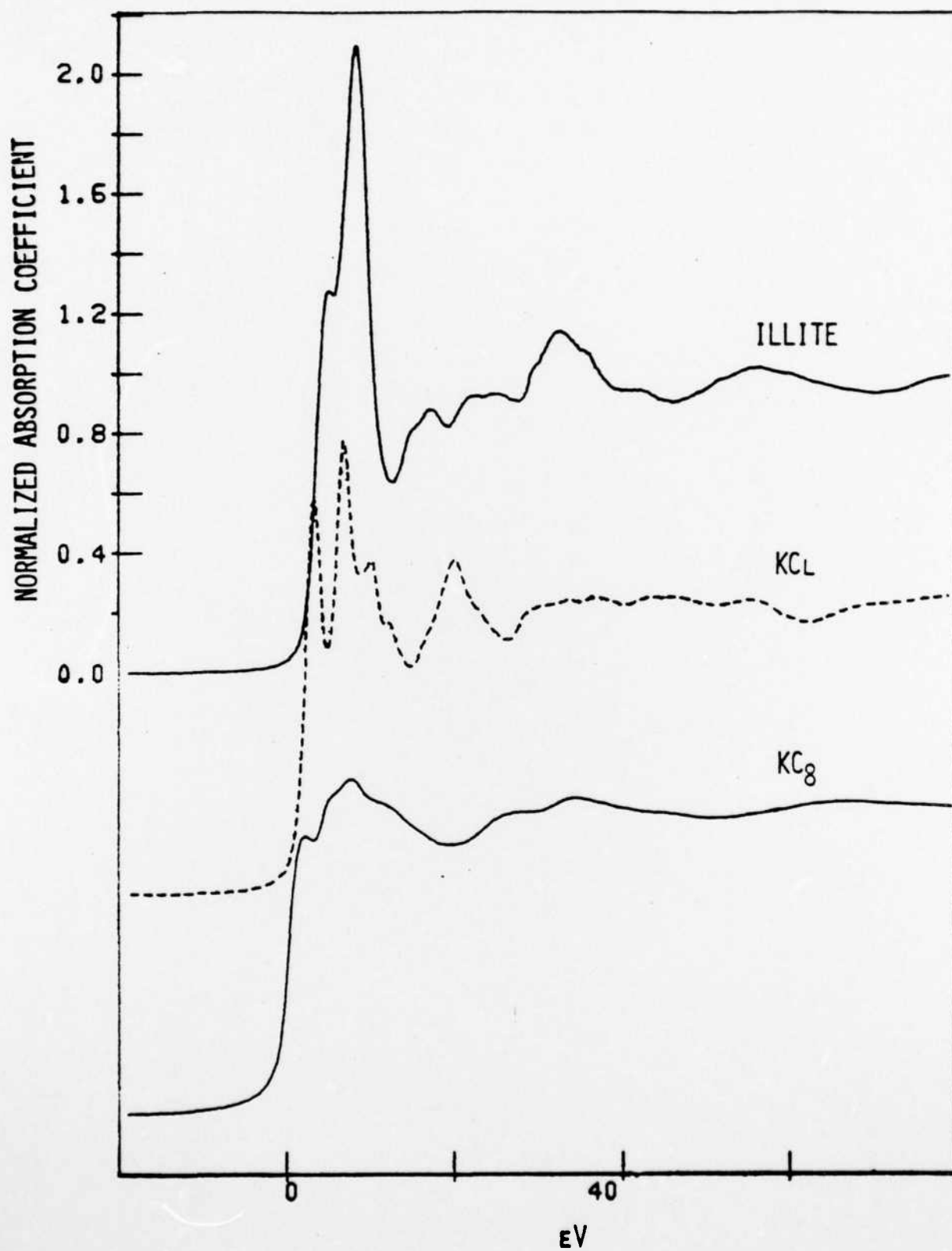


Fig. 8. K-edge spectra for potassium in selected compounds.

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